PRESOLAR SPINEL GRAINS FOUND IN FINE–GRAINED RESIDUE FROM THE MURRAY CM2 CARBONACEOUS CHONDRITE. E. Zinner[1], S. Amari[1], and R. S. Lewis[2], [1]Laboratory for Space Sciences and the Physics Department, Washington University, St. Louis, MO 63130, USA (ekz@wuphys.wustl.edu), [2]Enrico Fermi Institute, University of Chicago, Chicago IL 60637 USA.

Introduction: By far most of the presolar oxide grains identified so far are corundum and Al-Mg spinel constitutes only a small fraction [1-4]. Essentially all previous isotopic studies of single presolar oxide grains were made on grains of 1 µm or larger diameters. Zinner and Tang [5] measured O isotopic ratios in aggregates of grains from the Murray (CM2) acid residue CF, whose nominal grain size is <0.2 µm [6] and found variable excesses in ^{17}O. Most of the grains in Murray CF are Al-Mg spinel 0.1-0.2 µm in size [7]. If the ^{17}O excesses are due to presolar oxide grains with isotopic compositions that are, on average, the same as those of presolar corundum grains [1,2], ~2% of the Murray CF oxide grains should be presolar. Thus, either the fraction of presolar spinel grains in Murray CF is much higher than among larger spinel grains or presolar grains in CF are corundum but then most of these corundum grains would have to be of presolar origin.

A new type of ion microprobe, the NanoSIMS, with its high spatial resolution, high sensitivity, and multidetection capability [8] has made it possible to attempt the isotopic analysis of single oxide grains from Murray CF. Here we report the first O isotopic measurements of grains of this size.

Experimental: Murray CF, a size separate obtained after destruction of all silicates and carbon with HF-HCl, $Cr_2O_7^{2-}$, and $HClO_4$ [6], consists of ~80% spinel, the rest is mostly presolar SiC [7]. CF grains were deposited from liquid suspension onto a Au foil for isotopic analysis in the NanoSIMS. Terrestrial Al_2O_3 grains of 0.3 µm in diameter were also deposited as standards as were small grains of Murchison matrix.

Isotopic measurements were made in multidetection mode by counting secondary O^- ions, produced by Cs^+ bombardment, in three electron multipliers. The two remaining small electron multipliers were used to count $^{24}Mg^{16}O^-$ and $^{27}Al^{16}O^-$ ions in order to distinguish between Al-Mg spinel and other oxide grains. Grains to be analyzed were identified from 20µm×20µm raster images of secondary electrons and $^{16}O^-$ ions produced by the Cs beam. For an isotopic measurement, the primary beam was deflected onto the selected grain and rastered over a 0.6µm×0.6µm area. The taking of a raster image took 2 minutes, the isotopic analysis of a single grain only 25 seconds, and many CF grains were completely sputtered away during that time. Analyses of Murray CF grains were interspersed with measurements on the terrestrial Al_2O_3 standard used for calibration of the isotopic ratios. Since the detection efficiency varies from one electron multiplier to the next, such calibration is even more important than the correction for isotopic mass fractionation during measurements with a single detector in peak jumping mode. The Murchison matrix grains were used only to center on the $^{24}Mg^{16}O^-$ peak.

Results and discussion: We measured 328 CF grains. The large majority are Al-Mg spinels and we did not detect a single corundum grain. About 10% of the grains either have too low $^{24}Mg^{16}O^-/^{16}O^-$ and $^{27}Al^{16}O^-/^{16}O^-$ ratios or too high $^{24}Mg^{16}O^-/^{16}O^-$ ratios for being spinel. Lacking any other elemental information, we do not know their mineral identity. The normalized oxygen isotopic ratios of 237 grains whose analytical 1σ errors in $^{17}O/^{16}O$ and $^{18}O/^{16}O$ are less than 10% are plotted in Fig. 1 with their 1σ errors. Most grains have small ^{16}O excesses, consistent with the O isotopic composition of spinel in carbonaceous chondrites [e.g., 9, 10], or are isotopically normal. However, six grains, labeled A-F in the figure, have anomalies >2σ (we applied this criterion very conservatively) and must be of presolar origin. These six grains are Al-Mg spinels. All of them have ^{17}O excesses, two have deficits and one an excess of ^{18}O.

In Fig. 2 we compare the O isotopic ratios of these six spinel grains with the ratios of previously analyzed large (>1 µm) corundum and spinel grains, the majority of which are from ordinary chondrites [2-4 and unpublished data by L. Nittler]. As can be seen, the CF and previously analyzed large spinel grains have more normal $^{16}O/^{18}O$ ratios than corundum. Part of the reason for this probably lies in the fact that most presolar corundum grains were located by ion imaging of the $^{16}O/^{18}O$ ratio and presolar grains with almost normal $^{16}O/^{18}O$ ratios were missed by this method [2, 11]. In fact, three, and possibly five, of the presolar CF grains would not have been identified in this way. On average, the ^{17}O excesses of the CF grains are smaller than those of the corundum grains.

We identified at least six presolar grains out of ~210 spinel grains from Murray CF. This fraction is much higher than the fraction of presolar grains found among larger (>1 µm) spinel grains from the Tieschitz meteorite. Nittler et al. [2] found only one out of ~4000 imaged spinel grains. In contrast, ~1% of corundum grains in this size range are of presolar origin. However, if larger presolar spinel grains had the same O isotopic ratios as the presolar CF grains, the imaging search might have missed 80% of them. Even then, the fraction of presolar grains among CF spinels (6/210) is much higher than among larger Tieschitz grains (≤1/1000). On the other hand, Tieschitz might be an anomaly in this respect. Choi et al. [3, 4] detected one presolar spinel among 49 spinel grains from Bishunpur and Semarkona, a fraction comparable to the one we found in Murray CF spinels. This compares to 25 presolar corundum grains out of 334 from these two meteorites. All these analyses were made on "large" grains. Still, if we want to compare the abundance of presolar grains among spinel grains of different sizes and from different meteorites, we are severely hampered by the statistics of small numbers. It is clearly desirable to measure large numbers of grains from spinel separates down to very small grain sizes. The development of automatic measurement procedures for multidetection isotopic analysis of individual grains with the NanoSIMS will hopefully give us the capability to accomplish this.

Acknowledgments: We gratefully acknowledge the role of Tang Ming in preparing Murray grain separates. We thank Larry Nittler for providing unpublished O isotopic data on presolar corundum and spinel grains.

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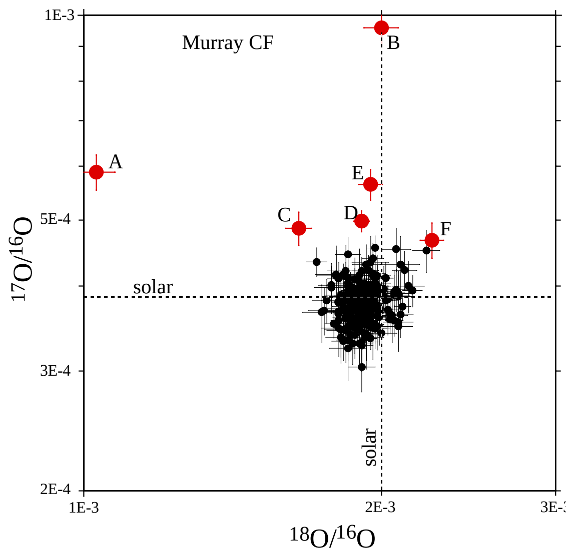

Figure 1. Oxygen isotopic ratios of 237 grains from the Murray CF separate. At least six of them have presolar isotopic signatures.

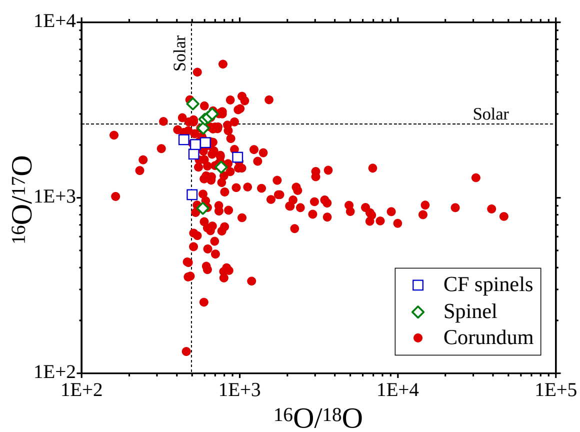

Figure 2. Oxygen isotopic ratios of presolar spinel grains are compared to those or previously measured corundum grains.